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(54) Fabric softening compositions

(57) The present invention relates to a liquid fabric softening composition which reduces the amount of dyes released from coloured fabrics upon wet domestic treatment such as those which occur in a laundry operation. The said composition comprises a cationic fabric softener having at least two long chains, a cationic dye fixing agent in amount of 0.1% to 10% by weight and is free of detergent materials.

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Description

Field of the invention

5 The present invention relates to fabric softening compositions and more particularly to compositions which reduce the amount of dyes released from coloured fabrics upon wet treatment such as those which occur in a laundry operation.

Background of the invention

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The domestic treatment of coloured fabrics is a problem known in the art to the formulator of laundry compositions. More particularly, the problem of formulating laundry compositions which reduce the amount of dyes released from coloured fabrics upon wet treatment is a particular challenge to the formulator. This problem is now even more acute with the trends of consumer to move towards more colored fabrics.

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Numerous solutions have been proposed in the art to solve this problem such as by treating the fabric with a dye scavenger during the washing process as described in EP 0,341,205, EP 0,033,815 and with a polyvinyl substance as described in WO 94/11482 or in the rinse cycle with a dye fixing agent as described in EP 0,462,806. However, a problem encountered with these solutions is that the dye fixing agents when used in the washing process may be destroyed or damaged by contact on storage and/or during the process, whilst when used in the rinse cycle the need for a detergent active and a high level of dye fixing agent is required to provide effective dye fixation performance. Furthermore, a problem related with the use of dye fixing agents in a softening composition is that of its weight efficiency. So that, although levels of dye fixing agents above 10% by weight would provide effective dye fixation, such use would result in an increase in the formulation cost. Another problem relating to the use of a high level of dye fixing agents in fabric softening compositions is that the resulting products show phase instability. On the other hand, lowering the level of dye fixing agents would not provide sufficient dye fixing properties and the need for a detergent material would be required.

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Accordingly, notwithstanding the advances in the art, there is still a need for a fabric softening composition which effectively reduces the amount of dyes released from coloured fabrics upon wet treatment.

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The Applicant has now found that the use of a liquid composition comprising a cationic fabric softener having at least two long chains, a cationic dye fixing agent in amount of 0.1% to 10% by weight, said composition being free of detergent materials, fulfills such a need.

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It is therefore an advantage of the invention to provide liquid fabric softening compositions which provide effective reduction of the amount of dyes released from coloured fabrics upon wet domestic treatments.

It is another advantage of the invention to provide liquid fabric softening compositions with effective softening properties.

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It is another advantage of the invention to provide liquid fabric softening compositions with effective storage stability properties.

For the purpose of the invention, the term "detergent materials" encompasses detergent surfactant materials selected from soaps, non-soap anionic, nonionic, zwitterionic and amphoteric synthetic and natural detergent surfactants which are not softeners. Not included by this definition are the fatty acids which are fabric softeners, contrary to said soaps components which do not have fabric softener properties.

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Summary of the invention

The present invention relates to a liquid fabric softening composition comprising a cationic fabric softener having at least two long chains and a cationic dye fixing agent in amount of 0.1% to 10% by weight, said composition being free of detergent materials.

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In accordance with another aspect of the present invention, a method for treating fabrics is provided. The method comprises contacting the fabrics during the rinse cycle of a consumer laundry process with an aqueous medium containing at least 50 ppm of a fabric softening composition of the invention.

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Detailed description of the inventionCationic fabric softeners

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An essential component of the invention is a cationic fabric softener component having at least two long chains. By component having at least two long chains is meant a component containing at least two alkyl or alkenyl chains, each comprising from 10 to 25 carbon atoms. Such fabric softener provides effective softness benefit to the treated fabrics.

Typical levels of said fabric softener components within the liquid softener compositions are from 1% to 80% by weight of the compositions. Depending on the composition execution which can be dilute with a preferred level of fabric

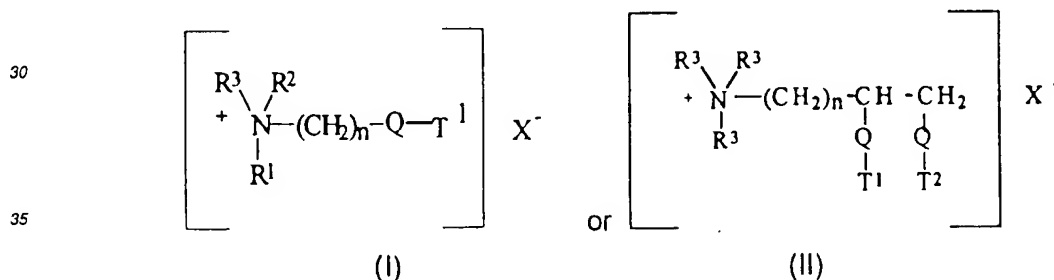
softening components from 1% to 5%, or concentrated, with a preferred level of fabric softening components from 5% to 80%, more preferably 10% to 50%, most preferably 15% to 35% by weight.

Typical cationic fabric softening components having at least two long chains include the water-insoluble quaternary-ammonium fabric softening actives, the most commonly used having been di-long alkyl chains ammonium chloride. Preferred cationic softeners among these include the following:

- 1) ditallow dimethylammonium chloride (DTDMAC);
- 2) dihydrogenated tallow dimethylammonium chloride;
- 3) dihydrogenated tallow dimethylammonium methylsulfate;
- 4) distearyl dimethylammonium chloride;
- 5) dioleyl dimethylammonium chloride;
- 6) dipalmityl hydroxyethyl methylammonium chloride;
- 7) stearyl benzyl dimethylammonium chloride;
- 8) tallow trimethylammonium chloride;
- 9) hydrogenated tallow trimethylammonium chloride;
- 10) C₁₂₋₁₄ alkyl hydroxyethyl dimethylammonium chloride;
- 11) C₁₂₋₁₈ alkyl dihydroxyethyl methylammonium chloride;
- 12) ditallow imidazolinium methylsulfate;
- 13) 1-(2-tallowylamidoethyl)-2-tallowyl imidazolinium methylsulfate.

However, in recent years, the need has arisen for more environmental-friendly materials, and rapidly biodegradable quaternary ammonium compounds have been presented as alternatives to the traditionally used di-long chain ammonium chlorides. Such quaternary ammonium compounds contain long chain alk(en)yl groups interrupted by functional groups such as carboxy groups. Said materials and fabric softening compositions containing them are disclosed in numerous publications such as EP-A-0,040,562, and EP-A-0,239,910.

The quaternary ammonium compounds and amine precursors herein have the formula (I) or (II), below :



wherein

Q is selected from -O-C(O)-, -C(O)-O-, -O-C(O)-O-, -NR⁴-C(O)-, -C(O)-NR⁴-;

R¹ is (CH₂)_n-Q-T² or T³;

R² is (CH₂)_m-Q-T⁴ or T⁵ or R³;

R³ is C₁-C₄ alkyl or C₁-C₄ hydroxyalkyl or H;

R⁴ is H or C₁-C₄ alkyl or C₁-C₄ hydroxyalkyl;

T¹, T², T³, T⁴, T⁵ are independently C₁₁-C₂₂ alkyl or alkenyl;

n and m are integers from 1 to 4; and

X⁻ is a softener-compatible anion.

Non-limiting examples of softener-compatible anions include chloride or methyl sulfate.

The alkyl, or alkenyl, chain T¹, T², T³, T⁴, T⁵ must contain at least 11 carbon atoms, preferably at least 16 carbon atoms. The chain may be straight or branched.

Tallow is a convenient and inexpensive source of long chain alkyl and alkenyl material. The compounds wherein T¹, T², T³, T⁴, T⁵ represents the mixture of long chain materials typical for tallow are particularly preferred.

Specific examples of quaternary ammonium compounds suitable for use in the aqueous fabric softening compositions herein include :

- 1) N,N-di(tallowyl-oxy-ethyl)-N,N-dimethyl ammonium chloride;

- 2) N,N-di(tallowoyl-oxy-ethyl)-N-methyl, N-(2-hydroxyethyl) ammonium chloride;
 - 3) N,N-di(2-tallowyl-oxy-2-oxo-ethyl)-N,N-dimethyl ammonium chloride;
 - 4) N,N-di(2-tallowyl-oxy-ethylcarbonyl-oxy-ethyl)-N,N-dimethyl ammonium chloride;
 - 5) N-(2-tallowoyl-oxy-2-ethyl)-N-(2-tallowyl-oxy-2-oxo-ethyl)-N,N-dimethyl ammonium chloride;
 - 6) N,N,N-tri(tallowyl-oxy-ethyl)-N-methyl ammonium chloride;
 - 7) N-(2-tallowyl-oxy-2-oxo-ethyl)-N-(tallowyl-N,N-dimethyl-ammonium chloride);
 - 8) N-methyl - N - (3 - tallowamidopropyl) , N-(2-tallowoyloxyethyl) ammonium chloride;
 - 9) 1,2-ditallowyl-oxy-3-trimethylammoniopropane chloride;
- and mixtures of any of the above materials.

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Of these, compounds 1-8 are examples of compounds of Formula (I); compound 9 is a compound of Formula (II). Particularly preferred is N,N-di(tallowoyl-oxy-ethyl)-N,N-dimethyl ammonium chloride, where the tallow chains are at least partially unsaturated. The level of unsaturation of the tallow chain can be measured by the Iodine Value (IV) of the corresponding fatty acid, which in the present case should preferably be in the range of from 5 to 100 with two categories of compounds being distinguished, having a IV below or above 25. Indeed, for compounds of Formula (I) made from tallow fatty acids having an IV of from 5 to 25, preferably 15 to 20, it has been found that a cis/trans isomer weight ratio greater than 30/70, preferably greater than 50/50 and more preferably greater than 70/30 provides optimal concentration. For compounds of Formula (I) made from tallow fatty acids having an IV of above 25, the ratio of cis to trans isomers has been found to be less critical unless very high concentrations are needed.

Other examples of suitable quaternary ammoniums of Formula (I) and (II) are obtained by, e.g. :

- replacing "tallow" in the above compounds with, for example, coco, palm, lauryl, oleyl, ricinoleyl, stearyl, palmityl, or the like, said fatty acyl chains being either fully saturated, or preferably at least partly unsaturated;
- replacing "methyl" in the above compounds with ethyl, ethoxy, propyl, propoxy, isopropyl, butyl, isobutyl or t-butyl;
- replacing "chloride" in the above compounds with bromide, methylsulfate, formate, sulfate, nitrate, and the like.

In fact, the anion is merely present as a counterion of the positively charged quaternary ammonium compounds. The nature of the counterion is not critical at all to the practice of the present invention. The scope of this invention is not considered limited to any particular anion.

By "amine precursors thereof" is meant the secondary or tertiary amines corresponding to the above quaternary ammonium compounds, said amines being substantially protonated in the present compositions due to the pH values.

For the preceding biodegradable fabric softening agents, the pH of the compositions herein is an essential parameter of the present invention. Indeed, it influences the stability of the quaternary ammonium or amine precursors compounds, especially in prolonged storage conditions. The pH, as defined in the present context, is measured in the neat compositions at 20°C. For optimum hydrolytic stability of these compositions, the neat pH, measured in the above-mentioned conditions, must be in the range of from 2.0 to 4.5. Preferably, where the liquid fabric softening compositions of the invention are in a diluted form, the pH of the neat composition is in the range of 2.0 to 3.0. The pH of these compositions herein can be regulated by the addition of a Bronsted acid. Examples of suitable acids include the inorganic mineral acids, carboxylic acids, in particular the low molecular weight (C₁-C₅) carboxylic acids, and alkylsulfonic acids. Suitable inorganic acids include HCl, H₂SO₄, HNO₃ and H₃PO₄. Suitable organic acids include formic, acetic, citric, methylsulfonic and ethylsulfonic acid. Preferred acids are citric, hydrochloric, phosphoric, formic, methylsulfonic acid, and benzoic acids.

Cationic dye fixative agent

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The other essential component of the invention is a cationic dye fixative agent. Cationic dye fixing agents, or "fixatives", are well-known, commercially available materials which are designed to improve the appearance of dyed fabric by minimizing the loss of dye from fabrics due to washing. Cationic dye fixatives are based on various quaternized or otherwise cationically charged organic nitrogen compounds. Cationic fixatives are available under various trade names from several suppliers. Representative examples include: CROSCOLOR PMF (July 1981, Code No. 7894) and CROSCOLOR NOFF (January 1988, Code No. 8544) from Crosfield; INDOSOL E-50 (February 27, 1984, Ref. No. 6008.35 84; polyethyleneamine-based) from Sandoz; SANDOFIX TPS, which is also available from Sandoz and is a preferred polycationic fixative for use herein and SANDOFIX SWE (cationic resinous compound), REWIN SRF, REWIN SRF-O and REWIN DWR from CHT-Beitlich GMBH.

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Other cationic dye fixing agents are described in "Aftertreatments for improving the fastness of dyes on textile fibres" by Christopher C. Cook (REV. PROG. COLORATION Vol. 12, 1982). Dye fixing agents suitable for use in the present invention are ammonium compounds such as fatty acid - diamine condensates e.g. the hydrochloride, acetate, metosulphate and benzyl hydrochloride of oleyldiethyl aminoethylamide, oleylmethyl-diethylenediaminemethosulphate, monostearyl-ethylene diaminotrimethylammonium methosulphate and oxidized products of tertiary amines; derivatives

of polymeric alkyldiamines, polyamine-cyanuric chloride condensates and aminated glycerol dichlorohydrins.

The amount of dye fixing agent to be employed in the composition of the invention is in amount of from 0.1% to 10% by weight of the composition, preferably from 0.5% to 8% by weight, more preferably from 0.8% to 5.5% by weight of the composition.

- 5 For optimum dye fixing benefit as well as softness benefit, the weight ratio of fabric softener to dye fixing agent is of from 60:1 to 1.5:1, more preferably from 20:1 to 3.5:1, most preferably from 10:1 to 3.5:1.

Detergent materials

- 10 For the purpose of the invention, the term "detergent materials" encompasses detergent surfactant materials selected from soaps, non-soap anionic, nonionic, zwitterionic and amphoteric synthetic and natural detergent surfactants which are not softeners. Not included by this definition are the fatty acids which are fabric softeners, contrary to said soaps components which do not have fabric softener properties.

- Soaps and non-soaps anionic materials are the linear and branched primary alkyl sulfates, alkyl ethoxysulfates, 15 fatty oleyl glycerol sulfates, alkyl phenol ethylene oxide ether sulfates, the C5-C17 acyl-N-(C1-C4 alkyl) and -N-(C1-C2 hydroxyalkyl) glucamine sulfates, and sulfates of alkylpolysaccharides; anionic sulfonate surfactants such as the salts of C5-C20 linear alkylbenzene sulfonates, alkyl ester sulfonates, C6-C22 primary or secondary alkane sulfonates, C6-C24 olefin sulfonates, sulfonated polycarboxylic acids, alkyl glycerol sulfonates, fatty acyl or oleyl glycerol sulfonates, alkyl ethoxy carboxylates, and the alkali metal sarcosinates.

- 20 Nonionic detergent materials are the polyhydroxy fatty acid amides, polyethylene, polypropylene, and polybutylene oxide condensates of alkyl phenols; alkyl ethoxylate condensation products of aliphatic alcohols with from about 1 to about 25 moles of ethylene oxide; ethoxylated C₆-C₁₈ fatty alcohols and C₆-C₁₈ mixed ethoxylated/propoxylated fatty alcohols; alkylpolysaccharides and the fatty acid amides.

- Zwitterionic and amphoteric materials are the amine oxides, the alkyl amphocarboxylic acids, the betaines such as 25 coconut acylamidopropyldimethyl betaine and hexadecyl dimethyl betaine.

Additional components

- The composition may also optionally contain additional components such as additional fabric softener materials, 30 electrolyte concentration aids, stabilisers, such as well known antioxidants and reductive agents, soil release polymers, bacteriocides, colorants, perfumes, preservatives, optical brighteners, anti ionisation agents, antifoam agents and chelating agents.

Additional fabric softener materials

- 35 Additional fabric softening materials may be used in addition to the di-long chain cationic fabric softener. When used, such additional fabric softening materials will typically be present in an amount of from 0 to 15% by weight of the composition. Such materials are the single long chain alkyl cationic softeners and/or the fatty acids.

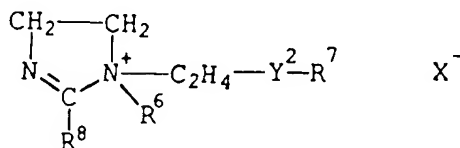
Single long chain alkyl cationic softeners

Such mono-long-chain-alkyl cationic softeners suitable for use herein are, preferably, quaternary ammonium salts of the general formula :



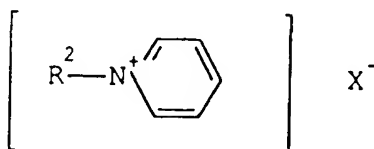
- wherein the R² group is C₁₀-C₂₂ hydrocarbon group, preferably C₁₂-C₁₈ alkyl group of the corresponding ester linkage interrupted group with a short alkylene (C₁-C₄) group between the ester linkage and the N, and having a similar hydrocarbon group, e.g., a fatty acid ester of choline, preferably C₁₂-C₁₄ (coco) choline ester and/or C₁₆-C₁₈ tallow choline 50 ester at from 0.1% to 20% by weight of the softener active. Each R³ is a C₁-C₄ alkyl or substituted (e.g., hydroxy) alkyl, or hydrogen, preferably methyl, and the counterion X⁻ is a softener compatible anion, for example, chloride, bromide, methyl sulfate, etc. Other cationic materials with ring structures such as alkyl imidazoline, imidazolium, pyridine, and pyridinium salts having a single C₁₂-C₃₀ alkyl chain can also be used. Very low pH is required to stabilize, e.g., imidazoline ring structures.

- 55 Some alkyl imidazolium salts and their imidazoline precursors useful in the present invention have the general formula :



wherein Y^2 is $-\text{C}(\text{O})-\text{O}-$, $-\text{O}(\text{O})\text{C}-$, $-\text{C}(\text{O})-\text{N}(\text{R}^5)-$, or $-\text{N}(\text{R}^5)-\text{C}(\text{O})-$ in which R^5 is hydrogen or a C_1 - C_4 alkyl radical; R^6 is a C_1 - C_4 alkyl radical or H (for imidazoline precursors); R^7 and R^8 are each independently selected from R^3 and R^2 as defined hereinbefore for the single-long-chain cationic surfactant with only one being R^2 .

Some alkyl pyridinium salts useful in the present invention have the general formula :



wherein R^2 and X^- are as defined above. A typical material of this type is cetyl pyridinium chloride.

25 Fatty acids

Suitable fatty acids include those containing from 10 to 25, preferably from 12 to 25 total carbon atoms, with the fatty moiety containing from 10 to 22, preferably from 16 to 22, carbon atoms. The shorter moiety contains from 1 to 4, preferably from 1 to 2 carbon atoms. The level of unsaturation of the tallow chain can be measured by the Iodine Value (IV) of the corresponding fatty acid, which in the present case should preferably be in the range of from 5 to 100, more preferably in the range of from 0 to 25.

Specific examples of fatty acid compounds suitable for use in the liquid fabric softening compositions herein include compounds selected from lauric acid, myristic acid, palmitic acid, stearic acid, arachidic acid, behenic acid, oleic acid, coconut fatty acid, tallow fatty acid, partially hydrogenated tallow fatty acid and mixtures thereof. A most preferred fatty acid compound is tallow fatty acid with an Iodine Value (IV) of 18.

When the above mentioned fatty acids are used, the fatty acid will be present in a weight ratio of said cationic fabric softening agents having di-long chains to said fatty acid compounds of from 25:1 to 6.5:1, more preferably from 20:1 to 10:1 and most preferably from 20:1 to 15:1. Indeed, if used outside of these ratios, the resulting product will tend to exhibit phase instability and/or viscosity problems.

40 Electrolyte Concentration Aids

Inorganic viscosity control agents which can also act like or augment the effect of the surfactant concentration aids, include water-soluble, ionizable salts which can also optionally be incorporated into the compositions of the present invention. Incorporation of these components to the composition must be processed at a very slow rate.

A wide variety of ionizable salts can be used. Examples of suitable salts are the halides of the Group IA and IIA metals of the Periodic Table of the Elements, e.g., calcium chloride, magnesium chloride, sodium chloride, potassium bromide, and lithium chloride. The ionizable salts are particularly useful during the process of mixing the ingredients to make the compositions herein, and later to obtain the desired viscosity. The amount of ionizable salts used depends on the amount of active ingredients used in the compositions and can be adjusted according to the desires of the formulator. Typical levels of salts used to control the composition viscosity are from 20 to 20,000 parts per million (ppm), preferably from 20 to 11,000 ppm, by weight of the composition.

Alkene polyammonium salts can be incorporated into the composition to give viscosity control in addition to or in place of the water-soluble, ionizable salts above. In addition, these agents can act as scavengers, forming ion pairs with anionic detergent carried over from the main wash, in the rinse, and on the fabrics, and may improve softness performance. These agents may stabilise the viscosity over a broader range of temperature, especially at low temperatures, compared to the inorganic electrolytes. Specific examples of alkylene polyammonium salts include L-lysine monohydrochloride and 1,5-diammonium 2-methyl pentane dihydrochloride.

Another ingredient is a liquid carrier. Suitable liquid carriers are selected from water, organic solvents and mixtures

thereof. The liquid carrier employed in the instant compositions is preferably at least primarily water due to its low cost relative availability, safety, and environmental compatibility. The level of water in the liquid carrier is preferably at least 50%, most preferably at least 60%, by weight of the carrier. Mixtures of water and low molecular weight, e.g., <200, organic solvent, e.g., lower alcohol such as ethanol, propanol, isopropanol or butanol are useful as the carrier liquid. Low molecular weight alcohols include monohydric, dihydric (glycol, etc.) trihydric (glycerol, etc.), and higher polyhydric (polyols) alcohols.

Form of the composition

The fabric softening composition can take a variety of physical forms including liquid such as aqueous or non-aqueous compositions.

Such compositions may be used as a rinse added product, or as a spray or foam product. Preferably, the present composition is in a rinse added form.

The compositions of the invention can be added directly in the rinse both to provide adequate usage concentration, e.g., at least 50 ppm and more preferably from 100 to 10,000 ppm of the liquid rinse added fabric softener compositions of the present invention.

Accordingly, a method is provided for treating fabrics comprising contacting said fabrics in the rinse cycle with an aqueous medium containing at least 50 ppm, preferably from 100 to 10,000 ppm of the liquid fabric softening composition of the invention.

Process

The fabric softening composition can conveniently be made according to well-known processes to the skilled person. An exemplary disclosure is given in EP-A-0,668,902.

The invention is illustrated in the following non-limiting examples, in which all percentages are on a weight basis unless otherwise stated.

In the examples, the abbreviated component identifications have the following meanings:

DEQA : Di-(tallowyl-oxy-ethyl) dimethyl ammonium chloride
 Fatty acid : Stearic acid of IV=18
 Electrolyte : Calcium chloride
 PEG : Polyethylene Glycol MW 4000

Example 1

The following fabric softening composition according to the present invention was prepared:

Component	A
DEQA	19.0
Hydrochlorid acid	0.02
Soil Release Polymer	0.2
PEG	0.6
Perfume	1.0
Electrolyte	1200ppm
Dye	50ppm
Sandofix TPS	5.0
Water and minors to balance to 100%	

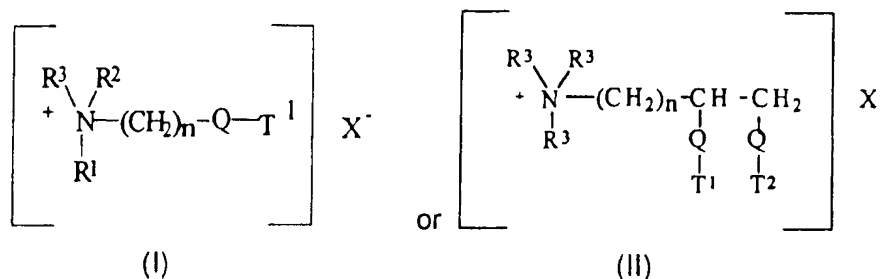
Example 2

The following fabric softening compositions are in accordance with the invention:

Component	B	C	D
DEQA	2.6	2.9	18.0
Fatty acid	0.3	-	1.0
Hydrochlorid acid	0.02	0.02	0.02
Soil Release Polymer	-	-	0.2
PEG	-	-	0.6
Perfume	1.0	0.5	1.0
Electrolyte	-	-	600ppm
Dye	10ppm	10ppm	50ppm
Sandofix TPS	1.6	1.6	5.0
Water and minors to balance to 100%			

Claims

1. A liquid fabric softening composition comprising a cationic fabric softener having at least two long chains and a cationic dye fixing agent in amount of 0.1% to 10% by weight, said composition being free of detergent materials.
2. A liquid fabric softening composition according to Claim 1, wherein said cationic dye fixing agent is a polycationic dye fixing agent.
3. A liquid fabric softening composition according to either one of Claims 1 or 2, wherein said cationic dye fixing agent is in amount of from 0.5% to 8% by weight, preferably from 0.8% to 5.5% by weight of the composition.
4. A liquid fabric softening composition according to any one of Claims 1-3, wherein said fabric softener is a biodegradable fabric softening material.
5. A liquid fabric softening composition according to Claim 4, wherein said biodegradable fabric softening material is selected from quaternary ammonium compounds and amine precursors having the formula (I) or (II), below



: wherein Q is selected from -O-C(O)-, -C(O)-O-, -O-C(O)-O-, -NR⁴-C(O)-, -

C(O)-NR⁴-;

R¹ is (CH₂)_n-Q-T² or T³;

R² is (CH₂)_m-Q-T⁴ or T⁵ or R³;

R³ is C₁-C₄ alkyl or C₁-C₄ hydroxyalkyl or H;

R⁴ is H or C₁-C₄ alkyl or C₁-C₄ hydroxyalkyl;

T¹, T², T³, T⁴, T⁵ are independently C₁₁-C₂₂ alkyl or alkenyl;

n and m are integers from 1 to 4; and

X⁻ is a softener-compatible anion.

6. A liquid fabric softening composition according to any one of Claims 1-5, wherein said fabric softener is present in amount of 1% to 80% by weight.
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7. A method for treating fabrics comprising contacting said fabrics in the rinse cycle with an aqueous medium containing at least 50 ppm of a liquid fabric softening composition according to any one of Claims 1-6.

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European Patent
Office

EUROPEAN SEARCH REPORT

Application Number
EP 96 87 0068

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.6)
X,D	EP-A-0 462 806 (UNILEVER PLC ;UNILEVER NV (NL)) 27 December 1991 * page 2, line 1 - line 7 * * page 2, line 22 - line 34 * * page 3, line 12 - line 18 * * page 3, line 37 - line 41 * * page 4, line 38 - line 41 * * claims 1,9; example 3A * ---	1-7	C11D3/00 C11D3/30 C11D3/26
X	EP-A-0 188 242 (HENKEL KGAA) 23 July 1986 * claims 1,11,13; examples * ---	1-7	
A	EP-A-0 459 211 (KAO CORP) 4 December 1991 * page 2, line 3 - line 5; claims 1,4,11; example 4 * ---	1-7	
A	DATABASE WPI Section Ch, Week 9141 Derwent Publications Ltd., London, GB; Class A87, AN 91-299898 XP002016867 & JP-A-03 199 472 (KAO CORP) , 30 August 1991 * abstract * -----	1-7	
The present search report has been drawn up for all claims			
Place of search THE HAGUE		Date of completion of the search 7 November 1996	Examiner Loiselet-Taisne, S
CATEGORY OF CITED DOCUMENTS X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons ----- & : member of the same patent family, corresponding document			

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